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A COMPARISON OF SEVEN METHODS FOR CONCENTRATING ORGANIC CHEMICALS FROM ENVIRONMENTAL WATER SAMPLES

by

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ABSTRACT

Because there are no quantitative analytical techniques for the complex organic matter that occurs in chlorinated water, it is not possible to directly determine the efficiency of techniques for isolating them. Seven methods capable of isolating gram quantities of organic matter from water samples were evaluated by determining the ability of each to recover a set of model compounds possessing a wide variation in polarity, functional groups, water solubility and molecular weight. No single method appeared to be superior overall, based on the recovery of the model solutes, but some could be eliminated from field application for the present time since the adsorbents required were not commercially available. Field application of two methods were undertaken and the samples collected are being tested in several bioassays.

INTRODUCTION

A major objective of the U.S. Environmental Protection Agency (EPA) is to estimate the health risk associated with organic matter in potable water. Such estimates are made using various biological tests, in which animals or lower organisms are exposed to the organic contaminants in drinking water at sufficiently high concentrations to insure that the lack of a positive response provides a desired margin of safety. Estimates can be made by assessing each chemical contaminant separately or by testing directly a concentrate of the aqueous sample of interest. A direct evaluation of the water sample is generally not possible because the concentration of organic matter in most drinking water sources is less than 10 mg/L. Because the organic matter in surface waters is a complex mixture of natural and anthropogenic substances that defies complete analytical characterization, the direct toxicological evaluation of organic concentrates offers a practical alternative.

Two general classes of methods can be functionally defined for preparing concentrates of organic substances. Concentration methods involve the removal of water (e.g. lyophilization, freeze concentration, vacuum distillation, reverse osmosis and ultrafiltration), resulting in a more highly concentrated aqueous solution of organic contaminants. Isolation methods are those methods in which the organic substances are physically removed from the aqueous solution, e.g., adsorption onto a solid substrate followed by desorption (1).

EVALUATION CONDITIONS

The approach taken by the Health Effects Research Laboratory of the

U.S. EPA to determine the most acceptable method for producing sample concentrates for biological testing was to solicit proposals for practical methods that could be used in the field to concentate/isolate organic matter 50-fold or more from water. Because potable waters are ever changing sources of organic matter, these sources were considered unsuitable for use in any methods evaluation. Therefore a decision was made to evaluate various approaches under standardized conditions. Consequently, the approach taken was to have each research group (Table 1) determine the efficiency of their method in recovering a set of model solutes from organic free water. Each group was responsible for obtaining their own organic free water and model compounds. The humic acid was supplied by the U.S. Environmental Protection Agency. The model solutes shown in Table 2 reflect a wide range of physical and chemical properties and were selected for the most part from the list of "consensus voluntary reference compounds", recommended by the Council on Environmental Pollutants (2). The solute concentrations listed in Table 2 reflect the predominance of humic substances among the organic contaminants of drinking water.

After developing acceptable analytical techniques and conducting laboratory scale recovery studies, each investigator was to use his method to isolate/concentrate at least 50 fold the complete mixture of model solutes from 500 liters of organic free water (i.e. distilled water or equivalent containing 70 ppm $NaHCO_3$, 120 ppm $CaSO_4$, and 47 ppm $CaCl_2 \cdot 2H_2O$). The inorganic matrix was used in an attempt to simulate Cincinnati potable water (3) and to minimize the effect that differences in water sources may have on the efficiency of a particular method.

METHODS EVALUATED

Concentration Method

The concentration procedure that was developed and evaluated was a reverse osmosis (RO)/Donnan dialysis system (4). The initial objective during method development was to conduct membrane screening tests to evaluate the suitability of various RO and ion exchange membranes. The four membranes considered for final evaluation based on solute rejection, chlorine stability, and artifact production were the cellulose acetate and FT-30® (Film Tec Inc.) RO membranes, the Nafion® cation exchange membrane and the IONAC MA 3475 anion exchange membrane.

The method basically involved repetitive batch concentration whereby 167L portions of water containing the model solutes were forced by applied pressure through a semi-permeable membrane against the osmotic pressure gradient. The RO system was operated in a recirculating mode so that the portion of the water not forced through the RO membrane was recycled back to the batch concentration tank along with the inorganic salts and model solutes rejected by the membrane. Because the inorganic salts are concentrated along with the model solutes, a 4 hour Donnan softening cycle was used to reduce the concentration of calcium ions by exchanging them with sodium ions, thus preventing inorganic salt precipitation.

Isolation Methods

One of the isolation methods evaluated was a liquid-liquid extraction procedure utilizing supercritical fluid (SCF) CO_2 as the extraction solvent (5). The effectiveness of SCF CO_2 as an extraction solvent as compared to gaseous and liquid CO_2 is associated with the marked increase

in the density of CO_2 at its critical temperature and pressure resulting in increased solvating power. The extraction unit that was evaluated was an open system consisting of a SCF CO_2 source, a stainless steel extraction vessel, a pressure reduction valve for CO_2 removal, and a U-tube trap system for solute collection.

The other isolation methods evaluated employed solid adsorbents to isolate the model solutes (6-8). The first of these used XAD-4, a macroreticular, polystyrene-divinylbenzene resin prepared by Rohm and Haas, into which trimethylamine groups had been introduced (9). The purpose of the resulting quaternary ammonium functional groups was to allow more efficient adsorption of acidic compounds without an appreciable loss of capacity for hydrophobic compounds. This is important because the vast majority of the organic matter in potable water is neutral or acidic in nature (10).

Desorption of the model compounds isolated on the quaternary XAD-4 (QXAD-4) resin column was accomplished by sequential elution with diethyl ether, methanol, diethyl ether, 0.1N HCl/ether, 0.1N HCl/methanol, and saturated HCl/methanol. The acidified organic solvents were used for acidic solute removal.

Evaluations of an integrated adsorption system were also conducted. In this sytem by varying the pH conditions the dissolved organics (model compounds) are separated into fractions by isolation onto Amberlite XAD-8, AG MP-50 cation exchange resin, and graphitized carbon black. The procedure is based on the separation of organic solutes into hydrophobic

and hydrophilic neutral, acidic, and basic fractions.

Another adsorption system evaluated was a high volume, high pressure, macroporous resin-based concentrator system designed to provide a 10,000-fold concentrate. This system used four stainless steel columns in series. Columns one through four were filled with AG MP-1 (Bio Rad), AG MP-50 (Bio-Rad), XAD-2 and XAD-7 (Rohm and Haas), respectively. Unlike the other adsorption processes, a pump system was employed for both the adsorption and desorption phase of the evaluation. Use of acetonitrile as the elution solvent permitted UV monitoring of the eluant.

Combination Methods

Combination methods refer to those methods that employ both concentration and isolation methodology. The modified parfait/distillation method that was evaluated used a series of adsorbents coupled with vacuum distillation to recover unadsorbed solutes (11). Porous polytetrafluoroethylene (PTFE) was used to adsorb the hydrophobic neutrals and in field sampling serves as a filter for particulate removal. Cation and anion exchange resins were then used to remove the ionized organic substances and to deionize the sample prior to the concentration of the nonadsorbed hydrophilic neutrals by vacuum distillation. Organic constituents were selectively desorbed from the ion-exchange resins using organic solvents containing either HCl or NH_3.

Direct solvent extraction of large volume water samples with immiscible organic solvents is generally not employed in the preparation of organic concentrates for biological testing. However, volume limita-

tions can be overcome by the use of a continuous liquid-liquid extraction (CLLE) apparatus in combination with a separate preconcentration technique such as reverse osmosis (12). While a RO preconcentration step was not actually performed, all test evaluations of the CLLE procedure were performed assuming a prior 15-fold concentration by RO. Consequently, evaluations were performed using higher concentrations of model solutes and inorganic salts. Use was made of a 1:10 solvent to water ratio and a solvent recycle feature to maximize solvent efficiency and to minimize solvent artifacts.

RESULTS AND DISCUSSION

The general goal of this overall effort, supported in part or full by the Health Effects Research Laboratory, was to develop an efficient method for preparing a 50-fold or greater concentrate that was representative of the organic constituents present in potable water. Ideally the procedure should avoid chemical transformations, be artifact free, have high capacity and result in minimal losses. In addition, the concentrates should be in a solvent or easily exchanged to a solvent system compatible with *in vivo* and *in vitro* biological test systems. Efforts to determine the most acceptable method for preparing representative concentrates based on recovery data and mass balance determinations were hampered by the fact that the different investigators varied the starting volumes, the final concentration factor and the total number of model solutes quantified because of their differing capabilities and problems unique to the individual method.

Model Compound Studies

Two major problems that each of the investigating teams faced

were how to effectively spike a large volume of water with a variety of model solutes of differing solubility and how to analyze the model solutes for mass balance determinations. Because aqueous solutions are not amenable to direct GC or GC/MS analyses, direct analytical testing of the aqueous influent or effluent in adsorption techniques and the aqueous phase in either RO or liquid-liquid extractions (LLE) was not possible. Therefore, liquid-liquid extractions became an integral part of all the mass balance determinations of the influent or feed stock. Additional analytical problems were associated with glucose, glycine, trimesic acid, quinaldic acid, humic acid and 5-chlorouracil none of which could be extracted by conventional LLE making mass balance determinations extremely difficult if not impossible.

However, recovery data were more easily obtained owing to the increased concentration of solutes and in some instances incorporation of an organic solvent in the desorption process that was compatible with GC analyses. The results of the different recovery studies are listed in Table 2. The original reports should be read for details and complete recovery data (4-8, 11, 12). Even though the recovery data indicate the shortcomings of preparing a representative concentrate of the organic contaminants in potable water, it does demonstrate certain trends concerning similar solutes and it does permit a limited comparison of methods.

Methods utilizing solid adsorbents, in general, as compared to RO and liquid-liquid extraction yielded higher recoveries for the majority of solutes. This was expected since a variety of ionic and nonionic adsorbents are now available for the specific recovery of acidic, basic

or neutral solutes. Like all concentration/isolation procedures, the use of solid adsorbents has certain limitations. Major problems generally associated with adsorbents are the potential introduction of artifacts, the separation of solutes into multiple fractions with different solvent properties, and the incompatibility of the various solvent systems with toxicological test systems. Possibly because of the extensive clean-up procedures used by each research group, artifact production was not shown to be a major factor. Even though fractionation is generally desirable for analytical purposes, the resultant increased costs of biological testing associated with multiple samples is often prohibitive. Consequently, one is faced with recombining the fractions into a common solvent or solvent mixture suitable for subsequent health effects testing. None of the solvent systems used in the adsorption procedures under evaluation were considered compatible for direct toxicological testing.

Model solutes recovered at levels in excess of 50 percent by two or more methods were 2,4-dichlorophenol, isophorone, biphenyl, 1-chlorododecane, 2,4'-dichlorobiphenyl, 2,2',5,5'-tetrachlorobiphenyl, anthraquinone, bis-2-ethylhexyl phthalate and quinoline. Except for anthraquinone and quinoline these solutes are all hydrophobic. It is important to note that porous PTFE effectively recovered stearic acid in addition to each of the hydrophobic solutes previously mentioned. The higher recoveries by porous PTFE are not attributed to a greater adsorptive capacity but rather to the ease with which the hydrophobic solutes can be desorped by conventional elution processes.

Each of the methods had trouble recovering the more highly water

soluble or volatile model compounds such as trimesic acid, furfural, glucose, glycine, caffeine, and methylisobutyl ketone. Quinaldic acid, an amphoteric substance with moderate water solubility was also poorly recovered by each method with the exception of the quaternary XAD-4 procedure.

Field Application

Toward the end of the method evaluation period, the requirement arose to produce samples of organic materials for toxicological testing from a pilot drinking water treatment plant. The toxicologists wanted two types of samples: (1) a concentrated aqueous solution of the organic material that could be used as drinking water for the experimental animals and (2) a highly concentrated sample in an organic solvent for *in vivo* and *in vitro* testing. There were five streams at the plant, one undisinfected and one receiving either chlorine, chloramine, ozone or chlorine dioxide. There was also a carbon treatment step followed by redisinfection on the chlorinated stream so there were seven sampling points. It was determined that 2000 gallons of water from each would be required to provide the amount of residue needed for the desired sensitivity of the toxicological assays. Four sets of samples were to be taken over a one year period, so the total volume of water to be processed was 56×10^3 gallons (21.2×10^4L). Because of the magnitude of this project, the practicality of the methods had to be considered as well as the ability to recover certain classes of chemicals. It is apparent from the preceeding discussion that none of the methods was clearly superior in recovering all of the model compounds. Some methods required special adsorbents that were not commercially available (QXAD-4) or were not available in sufficient quantity

(powdered teflon or graphitized carbon black) for this project. As already discussed some of the adsorption methods produced samples in several incompatable solvent systems or in solvents not suitable for use in biological tests.

The method chosen to provide an aqueous concentrate was reverse osmosis. The reverse osmosis procedure provided 50-fold aqueous concentrates containing almost all of the organic carbon (4). However, the removal of salt that was required to achieve a 400-fold concentrate without precipitation and without forming a hypertonic solution resulted in substantial losses of organic carbon as described by Smith et al., (13).

The method chosen to provide a sample concentrate in an organic solvent was an adsorption method using XAD-8 and XAD-2 resins with a subsequent acetone elution. The system used for the adsorption method is illustrated in Fig. 1. The adsorption columns contained 5L of XAD resin that was cleaned by exhaustive extractions with methylene chloride, acetone and methanol prior to use. The XAD-8 resin was placed before the XAD-2 resin to remove humic material which is reported to bind irreversibly to XAD-2 (14). Two thousand gallons of water were passed through each set of columns at 9-11 bed volumes/hr. Immediately prior to entering the columns HCl was added to bring the water to pH 2. Samples were taken for total organic carbon (TOC) analysis before and after each resin column to moniter the efficiency of removal. Columns were eluted separately with three bed volumes of acetone followed by 3 bed volumes of acetone containing HCl.

Table 3 contains the results of total organic carbon analysis obtain-

ed for one of the samples taken during one of the collection of the organics from the chlorine treated streams. The TOC content of the sample at zero time is elevated because the methanol used to clean and store the resin had not been completely removed by rinsing the columns with 50 gallons of the sample stream prior to beginning the sample collection. The results obtained thereafter indicate that within the first 500 gallons of sample either equilibrium was reached or only a select fraction of the organic chemicals was being removed. While the actual amount of organic carbon removed was different for each stream, the results for each followed this pattern.

Similar results have been observed for granular activated carbon columns used in water treatment and have been attributed to either biological or chemical oxidation of TOC within the column (15). Because only 67 hours was required to collect the samples it is unlikely that biological oxidation is responsible for the significant losses in TOC; decreased TOC levels most probably result from the fact that the majority of the organic material in the water is highly polar and only the nonpolar fraction is being adsorbed. This view is strengthen by the fact that the least efficient recovery is observed in the ozonated water. It is generally accepted that ozonation produces more polar organic by-products than the other disinfectants and this sample would contain no residual ozone to cause oxidative degradation within the column.

If constant loading is assumed during the collection period, the percent of organic carbon adsorbed can be calculated (Table 4). Results varied from 30 percent for the ozonated water to 50 percent for the

chloraminated sample. The residues obtained after evaporating the acetone eluates were analyzed for carbon content. The amount of organic carbon contained in the residues of the acetone eluates of each column were calculated and compared to the organic carbon content of the volume of the water sample passed through the columns. The actual fraction of organic carbon recovered varied from 11% to 39%. The eluates obtained with acidified acetone could not be used because of condensation products of acetone which formed during storage.

To estimate the size column required to isolate more of this polar material we used information published by Thurman et al. (15), who described an empirical relationship between aqueous solubility of organic compounds and capacity factors on XAD-8 resin. They defined capacity factor as the mass of solute sorbed on the resin divided by the mass of solute present in the void volume of the column at the 50% breakthrough point. Based on their data a substance having a capacity factor of 1000 will have a solubility of about 1×10^{-3} moles/liter. Examples of compounds having solubility in this range are methyl benzoate, 2,4,6-trichlorophenol, and chlorobenzene.

By using Thurman's empirical formula, it was calculated that a column of 45L of XAD-8 would be required to completely retain these compounds. This size column would require 135L of solvent for elution. Collecting five samples at once would require a pilot plant for distillation and extraction comparable in magnitude to the water treatment pilot plant being sampled. Therefore, a decision was made to utilize smaller columns at the risk of losing more soluble organic compounds.

CONCLUSION

Of the seven methods evaluated both in the laboratory and in a field application, it is clear that an ideal method for recovering sufficient representative samples of organic matter from water samples for toxicological testing has not yet been developed. However, of those methods evaluated, the use of solid adsorbents was the most efficient and showed the greatest potential in concentrating organics from potable water for biological testing.

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Table 1

Research Groups Involved in Methods Evaluation

Institute	Principal Investigator	Method Evaluated
Drexel University	I.H. Suffet	RO/CLLE
Arthur D. Little	D.J. Ehntholt	LLE/SCF CO_2
Gulf South Research Institute	J.K. Smith	RO/Donnan Dialysis
Georgia Institute of Technology	E.S.K. Chian	Solid Adsorbents (XAD-8, AG MP-50, GCB)
University of Illinois	J.B. Johnston	Solid Adsorbents (PTFE, MSC-1, A-162)
Envirodyne Engineers Incorporated	D.C. Kennedy	Solid Adsorbent (Quaternary XAD-4)
Los Angeles County Sanitation District	R.B. Baird	Solid Adsorbents (MD-1, MP-50, XAD-2, XAD-7)

Table 2

THE PERCENT RECOVERY OF THE MODEL COMPOUNDS BY THE DIFFERENT PROCEDURES

Model Compound	Concentration in Test Solution ug/L	XAD-8, AG MP-50, GCB n = 5 100 L each	Quaternary XAD-4 n = 1 500 L	RO/Donnan Dialysis n = 1 500 L
Stearic Acid	50	7.8 ± 9.1	9.6	3.2
Trimesic Acid	50	47.6 ± 19.8	22.3	ND
2,4-Dichlorophenol	50	11.3 ± 11.3	48.4	52.0
Quinaldic Acid	50	ND	96.4	ND
Isophorone	50	44.4 ± 3.3	55.2	NS
Biphenyl	50	57.0 ± 14.5	52.4	8.0
1-Chlorododecane	5	95.2 ± 3.0	42.0	49.0
2,6-ditert-Butyl-4-methylphenol	50	6.8 ± 11.6	45.2	2.0
2,4'-Dichlorobiphenyl	50	70.1 ± 10.3	46.9	0.7
2,2',5,5'-Tetrachlorobiphenyl	5	65.0 ± 14.8	28.7	16.0
Anthraquinone	50	59.9 ± 13.8	45.3	5.0
Phenanthrene	1	38.7 ± 6.9	44.6	NT
bis(2-Ethylhexyl) phthalate	50	65.3 ± 21.2	15.6	ND
Glucose	50	NA	NA	16
Furfural	50	ND	0.6	0
Quinoline	50	61.1 ± 25.7	21.7	12.0
5-Chlorouracil	50	27.8 ± 10.7	41.7	45.0
Caffeine	50	39.3 ± 32.3	0.6	53.0
Glycine	50	4.8 ± 1.9	ND	ND
Chloroform	50	ND	43.5	21
Methylisobutyl ketone	50	3.3 ± 2.7	25.6	41
Humic Acid	2000	34.0 ± 4.6	40.7	54

ND = not detected
NA = not analyzed

Table 2 (continued)

THE PERCENT RECOVERY OF THE MODEL COMPOUNDS BY THE DIFFERENT PROCEDURES

Model Compound	SCF CO_2 n = 3 10 L each	PFTE, MSC-1, A-162 n = 3 8 L	MP-1, MP-50, XAD-2, XAD-7 n = 5 500 L	RO/CLLE n = 2 12.5L
Stearic Acid	ND	98.4 ± 6.6[d]	36.3 ± 14.0	NA
Trimesic Acid	ND	22.9 ± 13.9	ND	NA
2,4-Dichlorophenol	26.3 ± 8.5	51.5 ± 25.6	76.2 ± 10.8	74 ± 8[a]
Quinaldic Acid	ND	NA	31.8 ± 9.0	NA
Isophorone	28.0 ± 5.6	25.3 ± 9.0	77.0 ± 15.3	88 ± 12
Biphenyl	16.3 ± 11.9	89.2 ± 8.1[b]	81.6 ± 16.4	50 ± 17
1-Chlorododecane	25.0 ± 11.5	70.4 ± 8.0[b]	76.5 ± 33.7	19 ± 3
2,6-ditert-Butyl-4-methylphenol	30.7 ± 13.6	91.6 ± 10.3[b]	37.8 ± 23.0	NA
2,4'-Dichlorobiphenyl	45.0 ± 4.6	79.1 ± 3.5[b]	72.4 ± 9.2	NA
2,2',5,5'-Tetrachlorobiphenyl	30.3 ± 7.2	84.7 ± 4.6[b]	75.1 ± 12.6	NA
Anthraquinone	31.6 ± 29.3	109 ± 9.0[b]	74.6 ± 22.9	NA
Phenanthrene	14.3 ± 3.2	NA	NA	NA
bis(2-Ethylhexyl) phthalate	29.7 ± 4.7	94.3 ± 13.5[b]	65.7 ± 34.9	NA
Glucose	ND	NA	ND	NA
Furfural	3.0 ± 2.6	35.6 ± 12.9	6.6 ± 1.8	NA
Quinoline	4.3 ± 7.5	67.4 ± 35.8	79.9 ± 26.5	25 ± 5
5-Chlorouracil	9.3 ± 4.0	NA	7.5 ± 32.3	NA
Caffeine	6.0 ± 5.6	8.4 ± 1.2	41.8 ± 4.7	NA
Glycine	ND	NA	3.3 ± 3.1	NA
Chloroform	ND	NA	67.9 ± 22.8	NA
Methylisobutyl ketone	4.7 ± 0.6	NA	26.8 ± 15.7	NA
Humic Acid	1.0 ± 0	NA	NA	NA

ND = not detected
NA = not analyzed
[a] n=4, without humic acid present
[b] recovered only from PFTE

Table 3

Typical TOC Data (Mg/L) by Resin Process

TOC Sampling Point	Gallons				
	0	500	1000	1500	2000
Influent	3.6	3.5	3.4	3.4	3.4
After XAD-8	11.0*	2.2	2.7	2.7	2.5
After XAD-2	30.0*	1.6	2.0	2.1	1.9

*Resin contaminated with MeOH even after pre-rinse with 50 gallons of water.

Table 4

Organic Carbon Recovery With XAD Columns From
Drinking Water Pilot Plant Samples
Mg/L Organic Carbon

	Undisinfected	Ozone	Chlorine Dioxide	Chloramine	Chlorine
Influent TOC[a]	3.4	3.3	3.3	3.4	3.4
TOC After XAD-8[a]	2.5	2.8	2.2	2.3	2.5
TOC After XAD-2[a]	1.8	2.3	1.8	1.7	1.9
Apparent % Adsorbed	47	30	45	50	44
Percent Recovered with Acetone	30	11	24	39	39

[a]Average of values obtained at 500, 1000, 1500 and 2000 gallons during a typical run.

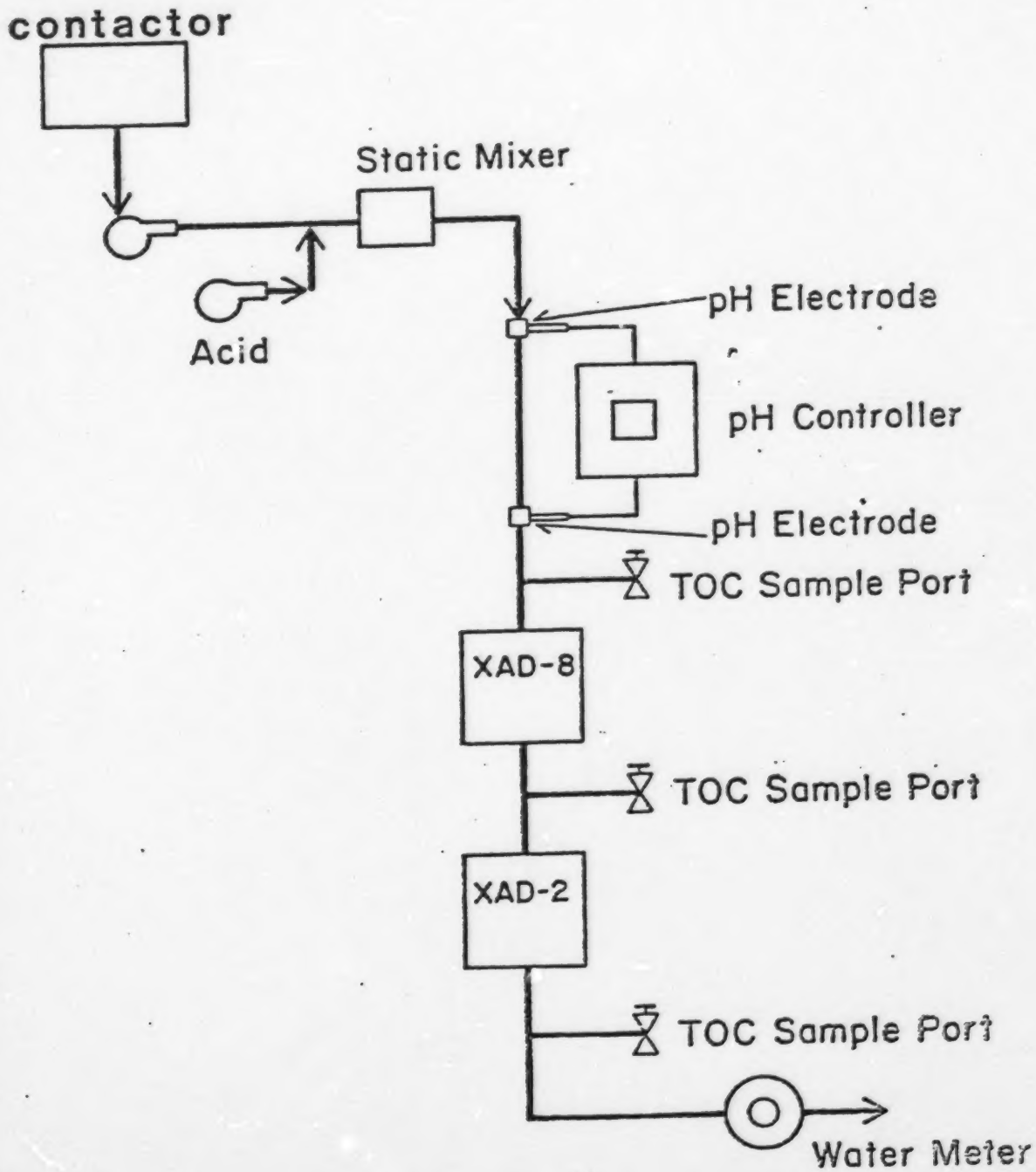

Figure 1. Schematic of adsorption process used for sample collection.